PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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Applicant(s):	Douglas R, Svenson, Jian Li	
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Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

May 5, 2009

DECLARATION OF DR. JIAN LI UNDER 37 C.F.R. § 1.132

Sir:

I, Jian Li, hereby declare and state that:

1. I am one of the inventors of the claimed invention of the above-identified U.S. Patent Application Serial No. 10/797,938, titled "Process for Manufacturing High Purity Xylose" (hereinafter referred to as "the Application"). I am currently employed by Rayonier, Inc. the assignee of the above-identified application, and have been at all times during and following the invention described by the Application.

2. I have studied and worked in the area of paper and pulp processes including pulp and fiber strength and have considerable experience in this field. Since December 2000 to the present time, I have been conducting research on the in-line control of the kraft pulping kinetics, and the physical chemistry of the alkaline hemicellulose solutions at Rayonier Research Center. I obtained a Ph.D.

degree in Chemical Engineering from McGill University, Montreal in December 1989; a Master of Engineering degree in Chemical Engineering from McGill University; and a Bachelor of Engineering degree from Tianjin Institute of Light Industry, China. From 1990 to 1997, I was employed with Pulp and Paper Research Institute of Canada (PAPRICAN), first as Assistant Scientist, then Associate Scientist, and eventually was promoted to Scientist. In December 1997, I joined the Institute of Paper Science & Technology in Atlanta, GA as an Associate Professor of Chemical Engineering where I conducted research and teaching until December 2000. A significant amount of my research during the 10 years I was at PAPRICAN and the Institute of Paper Science and Technology was directed towards the effect of hemicellulose change due to pulping, bleaching, and yield improvement on paper pulp fiber physical strength. I have published numerous articles in some of the most prestigious scientific journals in this research field, including *Journal of Pulp & Paper Science*; *TAPPI Journal*; *The Canadian Journal of Chemical Engineering*; *Chemical Engineering Science, Environmental Science & Technology*; and *Industrial Engineering Chemistry Research*. I have also presented conference papers in many of the most significant conferences in the Chemical Engineering and Pulp & Paper Science field, including the International Symposium on Wood and Pulping Chemistry, the CPPA Annual Conference and the TAPPI Pulping Conference. I am also a coinventor in 6 U.S. patents. A list of the various publications that I have authored or have coauthored is provided in the appendix.

3. I have reviewed the Office Actions dated March 25, 2008, including the basis of rejection of Claims 1 – 48 as being obvious over a combination of US Patent No. 6,512,110 to Heikkila et al. and U.S. Patent No. 4,294,654 to Turner.

4. I disagree with the assertions set forth in the Office Action dated March 25, 2008 that it would be obvious to one of ordinary skill in the art to substitute the

pulp used in the process for the production of xylose from hardwood pulp of the Heikkila patent with the prehydrolyzed pulp of Turner. In particular, as a person having a high level of skill in the art to which these patents pertain, I believe that the Examiner's rationale for asserting that it would be obvious to use Turner's pulp in the method/system of Heikkila is based on an incorrect or incomplete understanding of the teachings of these references, and is therefore incorrect.

5. Heikkila is concerned with a process for the preparation of xylose from a paper-grade hardwood pulp which includes treating the pulp with a xylanase enzyme treatment. See Abstract. Heikkila also repeatedly emphasizes that the process is directed to extraction of xylose from <u>paper-grade pulp</u>. See for example, Abstract; Column 1, line 8 – 12; column 5, lines 14 – 16; Examples 1 – 21 and the Claims. In particular, Heikkila emphasizes that the xylanase treatment "improves the quality of the pulp for use as a source of xylan in that it improves the solubility of xylan whereby xylan is more easily removed from pulp. This results in increased recovery of xylan and, accordingly, higher yields of xylose. Without being bound to the theory, these improvements are believed to have their basis in the increased solubility of xylan, which is caused by the xylanase treatment." See column 5, lines 5 – 13. A further objective of Heikkila's process is the production of a dissolving grade pulp. For example, Heikkila states "the xylanase treatment results in a simultaneous production of dissolving-grade pulp of very high quality and high yields of xylose." See column 5, lines 43 – 49, see also Column 9, lines 15 – 19.

6. Turner on the other hand is directed to a completely different and unrelated process than that of Heikkila. Specifically, Turner is directed to a process of delignification (i.e., removal of lignin from unbleached pulp) and bleaching of lignocellulosic pulp using a photo-oxygenation reaction. See column 2, lines 45 - 52. In particular, Turner teaches a process in which a stream of

oxygen gas is introduced into a lignocellulosic pulp slurry. See column 3, lines 51 – 53. The pulp slurry is then irradiated with ultraviolet light that delignifies and bleaches the lignocellulosic pulp. See column 4, lines 1 – 27. In other words, Turner teaches a process for removing lignin from prehydrolyzed pulp using oxygen and ultraviolet light.

7. One of ordinary skill in the art would not be motivated to combine Heikkila with Turner. First, Heikkila is directed to an entirely different field of application from the objective and purpose of Turner. As noted in paragraph 5, Heikkila is directed to a process of recovering xylose from paper grade pulp. This is quite different from the problem addressed by Turner, namely the delignification and bleaching of lignocellulosic pulp using oxygen with the aid of ultraviolet light. In fact, Turner is completely silent with respect to the recovery of xylose. Because of the significant differences to which these patents are directed, a person of ordinary skill in the art would not consider using the prehydrolyzed pulp mentioned by Turner in the xylanase enzyme treatment process of Heikkila.

8. Second, one of the preferred objectives of the process of Heikkila is to produce a dissolving grade pulp. That is, the end product of Heikkila's process is the simultaneous production of both xylose and a dissolving grade pulp. As such, one of ordinary skill in the art would not select the use of a dissolving grade pulp (i.e., the prehydrolyed pulp of Turner) as a starting product in the process of Heikkila. It would serve no purpose to modify the Heikkila process to include a desired end product as a starting product. Accordingly, one of skill in the art would not be motivated to modify the process of Heikkila to substitute a paper grade pulp with the prehydrolyed pulp of Turner.

9. Third, from the Examples in Turner, it can be seen that the untreated hardwood dissolving grade pulps (i.e., pre-hydroylzed pulp) of Turner have a relatively high lignin content as evidenced by the Permanganate Nos. See, for example, Table XIII. As is known in the art of pulping, the Permanganate No. of a pulp refers to the relative amount of lignin in the pulp. In particular, a Permanganate No. above 4.5 is indicative of a pulp having a lignin content, and in particular, a lignin content above 1 weight %. Additionally, if such high lignin content pulps are extracted with cold caustic extraction, significant amounts of lignin will be removed from the pulp along with the xylan. The resulting hemicaustic will contain the same order of magnitude of lignin and xylan. The resulting hemicaustic cannot be used to make xylose because it is contaminated with so much lignin. Further, separation of the lignin from xylan in the hemicaustic is technically not possible. Accordingly, one of skill in the art would recognize that the untreated pre-hydrolyzed pulps of Turner cannot be used in the process of Heikkila to arrive at the claimed invention because the resulting hemicaustic would have a high lignin content.

10. Fourth, the Examples in Turner also show that after treatment, the resulting pulps have relatively low viscosities. In particular, the treated hardwood dissolving grade pulps (i.e., pre-hydroylzed pulp) of Turner have low viscosities as evidenced in Table XIII. For instance, Runs1-4 all have viscosities of less than 14.8 cps. Pulp viscosity is a measure of cellulose chain length. With such low viscosity, these pulps cannot be used by dissolving pulp customers to make different products. Basically, Turner's process produces an unusable dissolving pulp for end users of the pulp. For instance, Run No. 4 of Table XIII has a viscosity of 5.7 cps. This pulp is a useless pulp since its viscosity is too low to be used for almost all dissolving pulp products. In addition to its useless nature for almost all dissolving pulp customers, more importantly, cold caustic extraction will extract significant amount of highly degraded cellulose along with xylan

from this pulp. As such, the organics in the resulting hemicaustic of this bleached pulp will contain significant amounts of short chain cellulose, which will convert to glucose during acid hydrolysis. In contrast, the claimed invention is directed to the use of a pre-hydrolyzed pulp having a low lignin content as well as a high xylan content so that a high recovery of xylose is possible. Such a xylose recovery is simply not possible with the pre-hydrolyzed pulps of Turner. Thus, the pulp of Turner cannot be used in the process of Heikilla to arrive at the claimed invention.

11. Finally, and as set forth in my Declaration of July 25, 2008, substituting a prehydrolyzed pulp for a paper grade pulp in the process of Heikkila would result in significant loss of pulp viscosity, which would render the resulting pulp unusable. The pulp viscosity, or IV, is a measure of the cellulose chain length, the higher IV, the longer the chain. For paper grade pulp, since it contains 15 - 20% short chain hemicellulose, the IV value of the pulp is a measure of the average of the mixture of cellulose and hemicellulose. Accordingly, when hemicellulose is removed from the pulp without degrading the cellulose, the resulting pulp will have a higher IV value than the starting pulp. In the case of the paper grade pulp used in the examples of Tables I – III, the cellulose chain should be greater than what is expected based on an IV of 7.97 (see column 8, line 41). As such, when hemicellulose is removed from the pulp without degrading the cellulose, it would be expected that the resulting pulp would have a higher IV because of the increase in cellulose concentration. However, based on the values shown in Tables I – III of Heikkila, the IVs of the treated pulp were predominately lower, and in some cases significantly lower, mostly because of the repeat treatment by the strong warm caustic and enzyme. This IV loss is probably fine when starting with a paper grade pulp with high IVs. However, a prehydrolyzed pulp after cooking has a relatively low IV already, generally below 7.0. As such, if a prehydrolyzed kraft pulp, such as the one described in Turner,

is treated in accordance with Heikkila's process, the resulting pulp would have an IV that would be too low to be used as a dissolving grade pulp. Accordingly, one of ordinary skill in the art would avoid using a prehydrolyzed pulp, such as that disclosed in Turner, in the process of Heikkila.

12. I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application of any patent issued thereon.

J. Li.

Jian Li Ph.D

Appendix

1) List of publications authored by Dr. Li

Publications authored by Dr. Li

1. Journal Publications

J. Samps, J. Li, "How does mass transfer affect the effectiveness of AQ", **Appita J.,** Vol 57 No 2, p.132-136, 2004.

C. Luthe, R. Berry, J. Li, "Polysulfide for yield enhancement in sawdust pulping: Does it work?" Pulp & Paper Canada, p. 32-37, 105(1), 2004

X-S. Chai, Q. Luo, J.Y. Zhu and J. Li, On-line Analysis of EA, Sulfide And Dissolved Lignin during Kraft Pulping Process by Attenuated Total Reflection UV Spectroscopy, **J. Pulp & Paper Sci**., 29(6), 204-207, 2003.

X-S. Chai, Zhu, J.Y., and Li, J., Simultaneous and Rapid Analysis of Hydroxide, Sulfide, and Carbonate in Kraft Liquors by UV-Attenuated Total Reflection Spectroscopy", **J. Pulp & Paper Sci.**, 28(4), 105-109, 2002.

X-S. Chai, Zhu, J.Y., and Li, J., An ATR-UV Sensor for Simultaneous Online Monitoring of Sulfide, Hydroxide, and Carbonate in Kraft Mill Recausticizing Operations, **J. Pulp & Paper Sci**., 28(4), 110-114, 2002.

J.Y. Zhu, X-S. Chai, X.J. Pan, Q. Luo, and J. Li, Quantification and Reduction of Organic Sulfur Compound Formation in a Commercial Wood Pulping Process, **Environ Sci. & Techno**., 36(10), 2269-2272, 2002.

S-H, Yoon, X-S. Chai, Zhu, J.Y., Li, J., and Malcolm, E.W., In-Digester Odor Reduction in Kraft Pulping," **Advances Environ. Res.**, 5(1), 91, 2001.

X-S. Chai, Yoon, S.-H., Zhu, J.Y., Li, J., The Fate of Hexenuronic Acid Groups during Pine Alkaline Pulping, **J. Pulp & Paper Sci**., 27(12), 407-411, 2001.

X-S. Chai, Q. Luo, Yoon, S.-H., Zhu, J.Y., and Li, J., The Fate of Hexenuronic Acid Groups during Hardwoods Kraft Pulping, **J. Pulp & Paper Sci**., 27(12),403-406, 2001.

X-S. Chai, Zhu, J.Y., and Li, J., A Simple and Rapid Method to Determine Hexenuronic Acid Groups in Chemical Pulps, **J. Pulp & Paper Sci**. 27(5), 165-170, 2001.

K. Goel, T. Radiotis, R. Eisner, G. Sherson, J. Li, "Switchgrass: a potential pulp fibre source,"**Pulp & Paper Canada**, 101:6, p.41-45, 2000.

J. Li, G. Moeser, L. Roen, "Nonuniformity of carbohydrate degradation during kraft pulping - measurement and modeling using a modified G-factor," **Ind. Eng. Chem. Res.**, vol.39, no. 4, p.916-921, 2000.

J. Li, M. MacLeod, R. Berry, J. McPhee, "Improving extended delignification technology for kraft pulping, Part II: Co-current two-stage continuous cooking ", **Pulp and Paper Canada**, vol. 100, No. 11, p.48, 1999.

J. Li, C. Mui, "Effect of lignin diffusion on kraft delignification kinetics as determined by liquor analysis. Part I: An experimental study", **J. Pulp & Paper Sci.**, vol.25, No.11, p.373, 1999.

T. Radiotis, J. Li, K. Goel, R. Eisner, "Fiber characteristics, pulpability, and bleachability of switchgrass," **Tappi J.**, vol. 82, No. 7, p.100, 1999.

Z. Li, H. Ma, G. Kubes, J. Li, "Synergistic Effect of Kraft Pulping with Polysulphide and Anthraquinone on Pulp Yield Improvement", **J. Pulp & Paper Sci.**, vol. 24, No.8, p.237, 1998.

J. Li, A. Phenix, M. MacLeod, "Diffusion lignin macromolecules within the fibre walls of kraft pulp. Part I: Determination of the restricted diffusion coefficient under alkaline conditions", **Canadian J. Chemical Engineering**, vol. 75, No.1, p.16, 1997.

J. Li, M.G. Paice, J.M. MacLeod, L. Jurasek, "The bleachability improvement of kraft pulp by alkaline leaching and xylanse treatment - their industrial implications", **J. Pulp & Paper Sci.**, vol.22, No. 6, p.J207, 1996.

J. Li, T. O'Hagan, M. MacLeod, "Using ultrafiltration to analyze the molecular weight distribution of kraft lignin at pH 13", **Canadian J. Chemical Engineering**, vol.74, Feb., p.110, 1996.

J. Li and A.R.P. van Heiningen, "Kinetics of solid state reduction of sodium sulfate by carbon", **J. Pulp & Paper Sci.**, vol.21, No. 5, p.J165, 1995.

J. Li, and A.R.P. van Heiningen, "The rate process of H_2S emission during steam gasification of black liquor char", **Chemical Engineering Science**, vol. 49, No. 24, p.4143, 1994.

J. Li and M. MacLeod, "Effect of alkaline leaching on pulp bleachability and physical properties", **Tappi J.**, vol. 76, No. 12, p.159, 1993.

J. Li and M. MacLeod., "Alkaline leaching of kraft pulps for lignin removal", **J. Pulp & Paper Sci.**, vol.19, No.2, Feb., p.J85, 1993.

J. Li and A.R.P. van Heiningen, "Kinetics of steam gasification of black liquor char", **Ind. Eng. Chem. Research,** vol.30, No.7, p.1594, 1991.

J. Li and A.R.P. van Heiningen, "Kinetics of gasification of fast pyrolysis kraft black liquor char by CO_2". **Ind. Eng. Chem. Research,** vol. 29, No.9, p.1776, 1990.

J. Li, and A.R.P. van Heiningen, "Sulfur emission during slow pyrolysis of black liquor char", **Tappi J**, vol.74, No.3., p.237, 1991.

J. Li and A.R.P. van Heiningen, "Sodium emission during pyrolysis and gasification of black liquor". **Tappi J**, vol.73, No.12, p.213, 1990.

J. Li and A.R.P. van Heiningen, "Kinetics of gasification of black liquor char by CO_2". **Canadian J. Chemical Engineering**, vol. 12, Aug., p.693, 1989.

J. Li and A.R.P. van Heiningen, "Kinetics of Na_2SO_2 reduction in the solid state by carbon monoxide". **Chemical Engineering Sci.**, vol. 43, No. 8, p.2079, 1988.

J. Li and A.R.P. van Heiningen, "The effect of sodium catalyst dispersion on the CO_2 gasification rate". **MRS Symposium series**, vol.111, MRS, p.441,1988.

J. Li and A.R.P. van Heiningen, "Mass transfer limitations in gasification of black liquor char by CO_2". **J. Pulp & Paper Sci.**, vol 12, No.5, p.J178, 1986.

2. Patents

J. Li, X.S. Chai, J. Zhu, "Simultaneous and Rapid Determination of Effective Alkali, Carbonate and Sulfide Concentrations in Kraft Liquors", **US patent No. 7,390,669 B2,** Jun 24, 2008.

J. Li, S. Boller, "Cellulosic fiber pulp and highly porous paper products produced therefrom", **US patent No. 7,285184 B2,** Oct. 23, 2007.

O. Ali, J. Cenicola, J. Li, J. Taylor, "Process for producing alkaline treated cellulosic fibers", **US Patent, No. 6,896,810,** May 24, 2005.

C. Luthe, J. Li, R. Berry, "Improved Pulping Process", **US Patent, No. 6,153,052,** Nov. 28, 2000.

J. Li, "Two stage kraft cooking", **US Patent, No. 5,522,958,** June 4, 1996.

A.R.P. van Heiningen, J. Li, and J. Fallavollita, "Low temperature recovery of kraft black liquor", **US Patent, No. 5,174,860**, Dec. 29, 1992. (= **Can. Patent, No. 1,313,577**, Feb. 1993.).

O. Ali, J. Li, S. Rogers, "Method of concentrating pulp mill extracts", **US patent application, No. 20,060,016,751,** Jan. 26, 2006

3. Representative Conference Publications

Li, J.; Moeser, G.; Rosen, L., "Non-uniformity of carbohydrate degradation during kraft pulping - measurement and modeling using a modified "G" factor," **the 10th International Symposium on Wood and Pulping Chemistry**, Yokohama, Japan, June 7-10, p.400, 1999.

Chai, X-S.; Zhu, J.; Li, J., "A simple and rapid method for determination of hexeneuronic acid." **the 10th International Symposium on Wood and Pulping Chemistry**, Yokohama, Japan, June 7-10, p.390, 1999.

Li, J.; MacLeod, J.M.; Berry, R., "Improving extended delignification technology for kraft pulping. Part IV: Strength and selectivity improvement with a low temperature/high alkali stage,' **the 10th International Symposium on Wood and Pulping Chemistry**, Yokohama, Japan, June 7-10, p.412, 1999.

J. Li, M. MacLeod, R. Berry, "Improving extended delignification technology for kraft pulping. Part III: pulp yield improvement from two-stage kraft cooking with initial polysulphide treatment," **Proceeding of TAPPI Pulping Conference**, Oct., Montreal, p.407-418(1998).

Z. Li, H. Ma, G. Kubes, J. Li, "Synergistic Effect of Kraft Pulping with Polysulphide and Anthraquinone on Pulp Yield Improvement", **Proceeding of 84th CPPA annual conference**, Montreal, Jan., p.B35—B39(1998).

Goel, K.; Eisner, R.; Sherson, G.; Radiotis, T.; Li, J., "Switchgrass: Potential Pulp-Fibre Source", **Proceeding of 84th CPPA annual conference**, Montreal, B109-114 (January 30, 1998).

J. Li, M. MacLeod, R. Berry, J. McPhee, "Improving extended delignification technology for kraft pulping, Part II: Co-current two-stage continuous cooking ", **Proceeding of 84th CPPA annual conference**, Montreal, Jan., p.B9-B14(1998).

J. Li, M. MacLeod, J. McPhee, R. Berry, T. O'Hagan, B. Thomas, B. Tristram, R. Kowalska "Improving extended delignification technology for kraft pulping, Part I:

Papri-Batch Cooking", **Proceeding of TAPPI pulping conference**, San Francisco, Oct., p.577(1997).